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Running title: FRACTOGRAPHY OF STEEL

Microstructure, mechanical properties and fractography of a manganese steel

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Abstract

A Fe-8.46%Mn-0.24%Nb-0.038%C (wt.%) manganese steel was investigated. The steel has a 100% bcc structure after heat treatment at 850°C for 1.5 h, water quenching or air cooling. Martensite interlocked microstructure consisting of fine martensite plates/needles with different spatial orientations was found. Austenite forms, in small amounts, after a

600°C reheating treatment. Scanning electron microscopy images and energy dispersive spectrometry of the fracture surfaces revealed both ductile and brittle types of failure and precipitates. Deep quenching after the heat treatments does not change the phase composition or the hardness. NbC is formed in the steel, in high number densities. It plays a role in the impact fracture process, by acting as void nucleation sites, facilitating ductile fracture with dimples appearing on the fracture surface.

Keywords: electron microscopy; hardness measurement; mechanical characterization; steel; fracture; X-ray diffraction

INTRODUCTION

The main role of nickel is to stabilise the austenitic structure in steel, which has relatively high ductility. Specific advantages of nickel include constant magnetic permeability, and very slight thermal expansion. It is very often used as an alloying element in iron alloys [1].

Huge industrial demand for nickel and nickel alloys means that the price of nickel has massively increased in recent years. An ability of manganese is to similarly stabilise the austenite in steel, as does nickel [2]. Outstanding toughness and high strain hardening capacity are the principal virtues of manganese steel. These properties are advantageous in heat-treated steels specified by mechanical engineers. This property of increasing hardenability rate is used to significant advantage, depending on the steel type and the end product, to improve mechanical properties. Manganese plays an important role as it lowers the temperature at which austenite transforms [3,4], thus refining the resulting pearlitic structures. When the cooling process is accelerated by quenching, austenite transforms into structures with high strength such as bainite and martensite.

Although the composition of manganese steel is extremely important in determining its properties, the heat treatment to which it is subjected to develop its great toughness or ductility can be even more so [4-6]. For the optimum development of strength, most steels are first fully converted to martensite. To achieve this, the steel must be quenched at a rate sufficiently high to avoid the decomposition of austenite during cooling to such products as ferrite, pearlite and bainite.

When the Mn content is greater than 5% (wt.%, throughout this paper), the hardness of steel increases, and it is also related to the presence of precipitates. Upon cooling after heating to 750-950°C, Mn significantly reduces the toughness of steel. The increase of σ -phase fraction and formation of MnS within manganese steels reduce their resistance to pitting corrosion. In addition, to obtain an $\alpha + \gamma$ two-phase structure, the required Mn, Ni, N element contents are relatively small. The cost of steel is relatively low at the same time, and steel has some features of $\alpha + \gamma$ duplex stainless steel. When more than 13% Mn is added, in the so-called high manganese steel, it becomes both hard and very ductile, and the lower critical cooling rate of steel improves the steel hardenability.

Sha et al. [1] have reported the hardness and impact energy of two experimental manganese alloys, Fe-19.7%Mn and Fe-19.7%Mn stabilised with 0.056%C-0.19%Ti-0.083%Al, under various heat treatment conditions. Further research is reported on a different experimental manganese steel in this paper, in order to find an appropriate way of heat treatment to improve the properties of the steel. This new steel has 8% Mn, and thus more economical than the previously studied 20% Mn steels.

MATERIALS AND METHODS

A manganese steel after different heat treatments was studied through scanning electron microscopy (SEM), X-ray diffraction (XRD), and impact and hardness testing. The steel composition is Fe-8.46%Mn-0.24%Nb-0.038%C.

HEAT TREATMENT AND MECHANICAL TESTING

For heat treating, steel specimens were firstly put into a furnace at 850°C for 1.5 hours, and then quenched by cool water (WQ) or air cooled (AC). Next, half of the specimens were put into furnace at 600°C for 4 hours this time, and then quenched by cool water or air cooled again. The treatment at 600°C was meant to introduce reverted austenite into the microstructure [1]. Some reverted austenite will transform to massive martensite and possibly epsilon martensite on WQ and AC from 600°C.

For Charpy impact toughness testing, heat treatments were carried out at Sheffield Metal Testing. Subsize Charpy V-notch samples with 10 mm by 5 mm by 55 mm cross-section were used. The temperatures of impact tests were room temperature, -20, -40, -50, -120, -140 and -150°C. Hardness measured on Rockwell C was obtained at Stocksbridge Engineering Steels. The Rockwell C hardness was then converted to Vickers hardness HV using the standard conversion given in ASTM standard E140 [7].

SCANNING ELECTRON MICROSCOPY AND X-RAY DIFFRACTION

The mounted steel specimen was ground using a grinding machine with 240, 500, 800, 1200 and 2500 grit sand paper successively. A polishing machine was operated with a colloidal

silica suspension to polish the ground steel specimen. Etching was performed by dipping cotton buds into a 5% hydrochloric acid (HCl) in alcohol solution, and swabbing the steel surface for 5 minutes.

For examining fracture surfaces, a hacksaw was used to cut the impact fractured specimens into the desired length (10-15 mm) without touching the fracture surface. The specimen also had to be cut perpendicular to the length of specimen, and had a flat surface to be glued vertically onto an aluminium disk. Methanol and acetone were used to rinse the fracture surface of each specimen.

The microstructure of the steel samples was examined in a JEOL 6500 field emission gun scanning electron microscope. The fracture surfaces of the samples were examined under the SEM to determine whether the sample surface had brittle cleavage type or ductile type failure. The etched surfaces of samples were analysed under SEM and energy dispersive spectrometry (EDS) to identify the precipitates and phases, where EDS was used with SEM for compositional microanalysis.

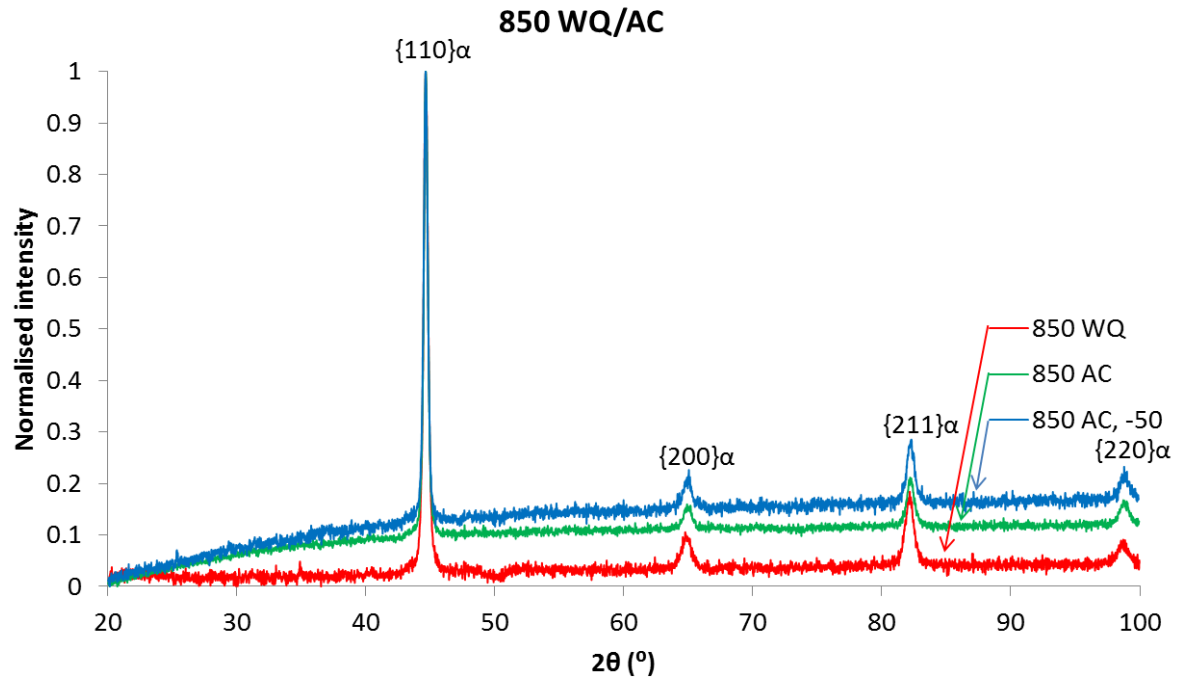
The X-ray diffraction analysis was performed using a PANalytical PW 1130 (40 kV, 40 mA) diffractometer equipped with Cu K α radiation. Diffraction patterns were taken in the range of 2θ from 20° to 100° .

RESULTS AND DISCUSSION

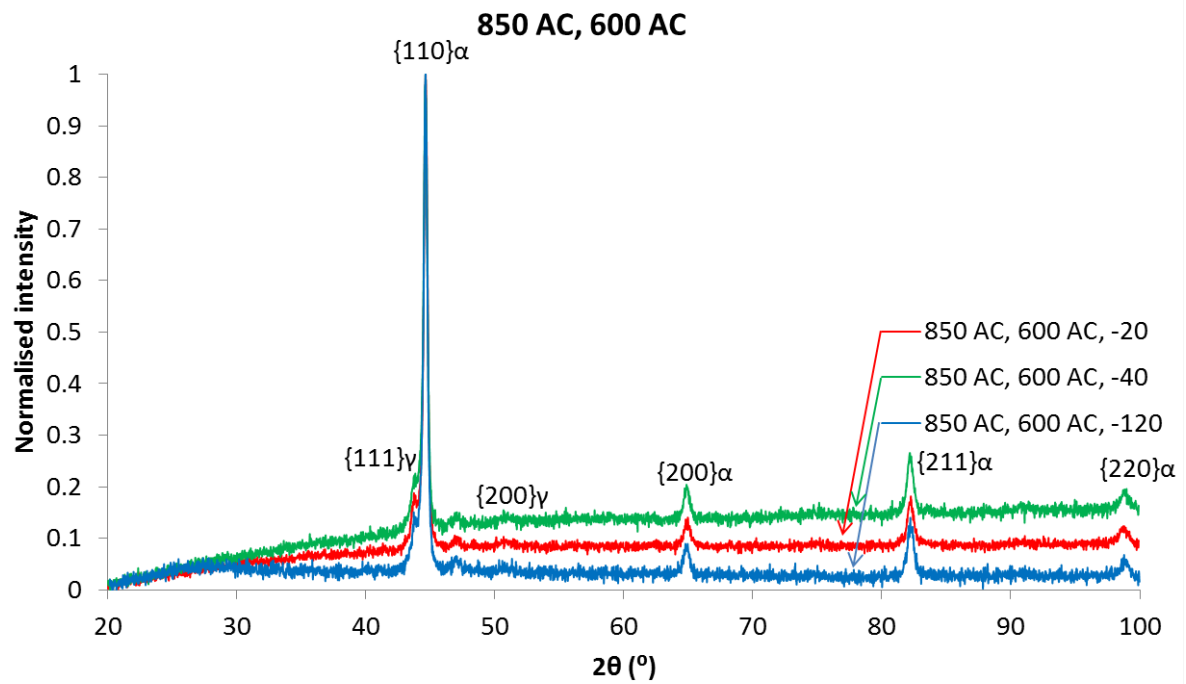
MICROSTRUCTURE

Fig. 1 shows the X-ray diffraction patterns of two groups of steel specimens, without (Fig. 1a) and with (Fig. 1b) the 600°C reheating treatment. It should be noted that the XRD specimens taken from the ends of the impact specimens had experienced deep quenching, to the negative temperatures indicated in the legends in Fig. 1, but not the impact stress. The purpose of carrying out XRD on impact tested specimens was to examine the effect of deep quenching on the phase composition. The major peaks are martensite peaks. Based on the composition of the alloy, it can be easily noticed that the carbon content is lower than 0.2%. Therefore, bcc martensite will not exist in those specimens [8]. It was found by XRD that the steel has a 100% bcc structure after 850°C 1.5 h, WQ or AC. Austenite only forms after the 600°C reheating treatment, by comparing Fig. 1a with Fig. 1b.

After heat treatment (both 850°C for 1.5 h, WQ and 850°C for 1.5 h, WQ, 600°C, WQ), some cracks were found on the steel blocks, after subsequent cutting. The main reason for the obvious cracks only appearing at the edge of the blocks could be the releasing of internal stress during the cutting [9,10].



(a)



(b)

Fig. 1. X-ray diffraction patterns of the steel after different heat treatments and impact test temperatures. (a) Without 600°C treatment; (b) with 600°C treatment. The heat treatment and cooling conditions are indicated in legends, where the last negative number gives impact test temperature.

Since martensite has a lower density than austenite, transformation between them results in a change of volume. When steel is quenched these volume changes occur very rapidly and unevenly throughout the specimen. The outside of specimen cools more quickly, and is mainly martensitic. In this case, expansion occurs. Internal stresses from this expansion generally take the form of compression on the crystals of martensite and tension on the remaining phase, with a fair amount of shear on both constituents. If quenching is done improperly, the internal stresses can cause a part to shatter as it cools. At the very least, they cause internal work hardening and other microscopic imperfections. It is common for quench cracks to form when water quenched, although they may not always be visible.

The typical martensite interlocked microstructure consisting of fine martensite plates/needles with different spatial orientations was found in the steel after the heat treatment of 850°C 1.5 h, WQ + 600°C 4 h, WQ and impact test at -120°C (Fig. 2). Two different types of microstructures were observed in the same specimen under higher magnification (Fig. 3) by SEM.

Though the low temperature phase is referred to as martensite when describing the steel in this paper, because their carbon content is virtually zero, the martensite behaviour is rather different from martensite in normal carbon steels. The martensite here is more akin to ferrite, and may be referred to as acicular-ferrite.



Fig. 2. Fe-8.46%Mn-0.24%Nb-0.038%C, 850°C 1.5 h, WQ + 600°C 4 h, WQ, and then impact tested at -120°C, showing martensite with different spatial orientations, which are acicular packets.

PRECIPITATES

The precipitates with different treatments were investigated by using SEM/EDS, including etched surfaces and fracture surfaces. The roles of niobium as an alloying element in steel are both its effect in solution and also, its ability to stabilise carbon by forming fine precipitates, whereby it effectively increases the steel strength through grain refining [11], retardation of recrystallisation, and precipitation strengthening. For example, niobium increases yield strength by precipitation hardening. The magnitude of increase depends on the size and amount of precipitated niobium carbide (NbC) [12]. Niobium atoms can pin dislocations both as a solute and after forming precipitates.

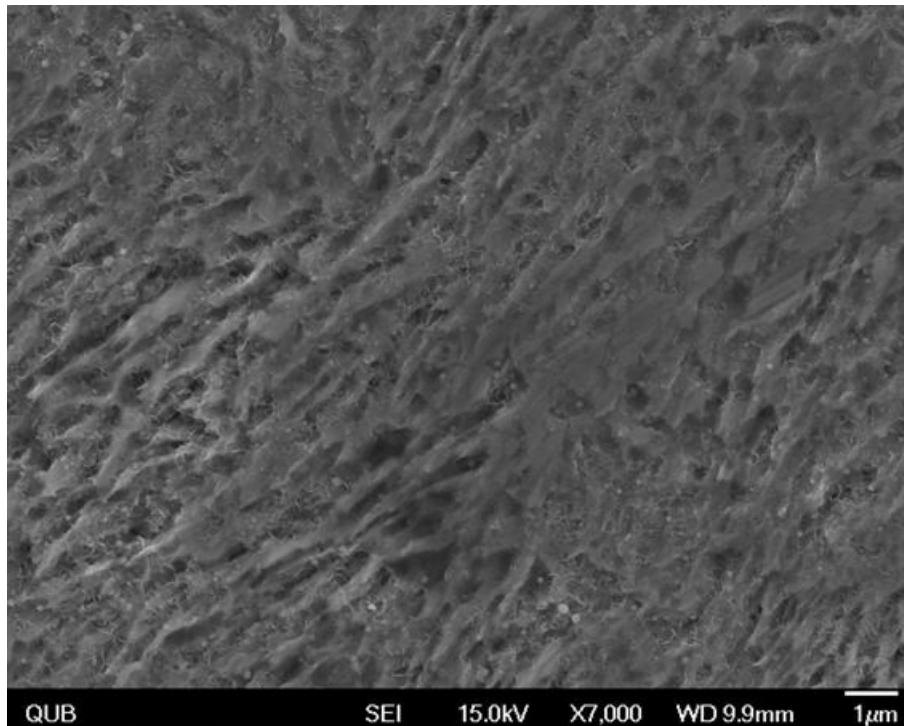


Fig. 3. Fe-8.46%Mn-0.24%Nb-0.038%C, 850°C 1.5 h, WQ + 600°C 4 h, WQ, and then impact tested at -120°C, at higher magnification, showing two types of structures, at two different size scales. The small size structures are in the nano range, and appear thin and flaky. Note that these are different from the appearance of martensite structures in mild steels.

NbC particles displayed a globular or cubic form and were observed on the etched surface, as shown in Fig. 4. NbC is a frequent intentional product in microalloyed steels, due to its extremely low solubility product in austenite, the lowest of all the refractory metal carbides [13]. This means that the sub-micrometre-sized precipitates of NbC are virtually insoluble at all processing temperatures and their location at grain boundaries helps prevent excessive grain growth in the steel. This is of enormous benefit and the cornerstone of microalloyed steels, because it is their uniform very fine grain size that ensures both toughness and strength. The fine grain size is a mechanism for strengthening. Finer grain size occurs in Nb steels, but precipitation and age hardening play an important role, too.

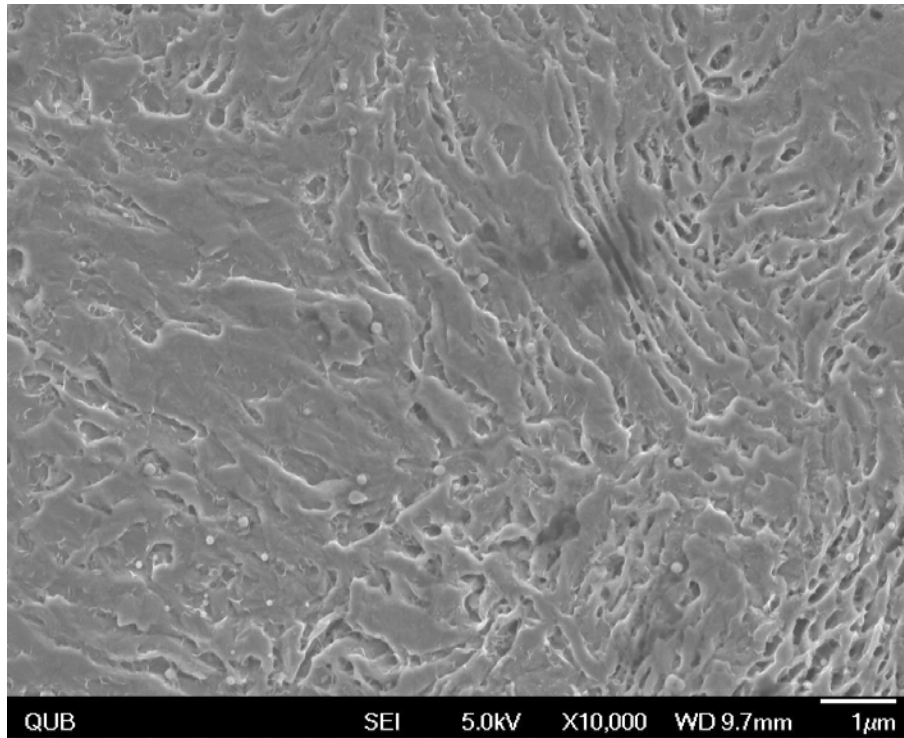


Fig. 4. Etched surface of Fe-8.46%Mn-0.24%Nb-0.038%C, 850°C 1.5 h, AC, showing niobium carbide, identified by EDS, precipitates, in globular or cubic form.

HARDNESS

Hardness was measured off each broken Charpy specimen (4-12 measurements for each condition), but the hardness results show small and apparently random fluctuation as a function of impact test temperature. The average hardness for individual conditions is given in Table 1. The reheating, at 600°C, does not appear to have an effect on hardness, comparing hardness of the steel with and without the 600°C treatment. Because the impact test temperature, i.e., deep quenching temperature, does not appear to affect the phase composition or the hardness, hardness data could be averaged over different impact specimens tested at different temperatures. The hardness averaged this way is included in Table 1.

Table 1. Hardness under different heat treatment and impact test conditions

Heat treatment	Impact test temperature (°C)	HV
850°C 1 h, AC	No impact test	277±6
	Various (-150 to room temperature)	279±1
850°C 1.5 h, WQ; 600°C 4 h, WQ	Room temperature	257±9
	-120	268±4
	Various (-140 to room temperature)	265±8
850°C 1.5 h, AC; 600°C 4 h, AC	-120	279±4
	Various (-120 to room temperature)	281±9

It is well known that hardening of steels requires heating to an austenitic phase and quenching to low temperature to produce a hard martensitic phase. A higher proportion of martensite structure should give higher hardness. In general, the Vickers hardness of martensite should be higher than that of ferrite. As the carbon content in the steel is very low, it would be very difficult to determine carbon contents, using, for examples, wavelength dispersive spectroscopy or transmission electron microscopy and energy dispersive spectroscopy.

CHARPY IMPACT FRACTOGRAPHY AND DISCUSSION ON TOUGHNESS

Figs. 5 and 6 show the SEM micrographs of fracture surface of the steel, after, respectively, 850°C 1.5 h, WQ + 600°C 4 h, WQ and 850°C 1.5 h, AC + 600°C 4 h, AC, and impact tested at -120°C. Two different types of fracture failure, cone shaped dimples (ductile) and honeycombs (relatively brittle), were observed in the two specimens, due to different cooling methods (water quenching and air cooling). It is clearly shown that the higher

impact energy, the more the ductile failure could be revealed, by comparing micrographs and results of impact test.

Fig. 7 shows a higher magnification SEM micrograph of the small crack propagating on the impact fracture surface of Fe-8.46%Mn-0.24%Nb-0.038%C, 850°C 1.5 h, WQ + 600°C 4 h, WQ, and then impact tested at -120°C. It was identified by EDS that the fractured precipitate was NbC. Generally, if yield strength is noticeably promoted, the critical crack size will be decreased and steels become sensitive to small flaws such as NbC and inclusions. Inclusions of about 0.1 μm would be enough to initiate the brittle cleavage fracture on the rigid zone ahead of the V-notch, where the stress is concentrated [14].

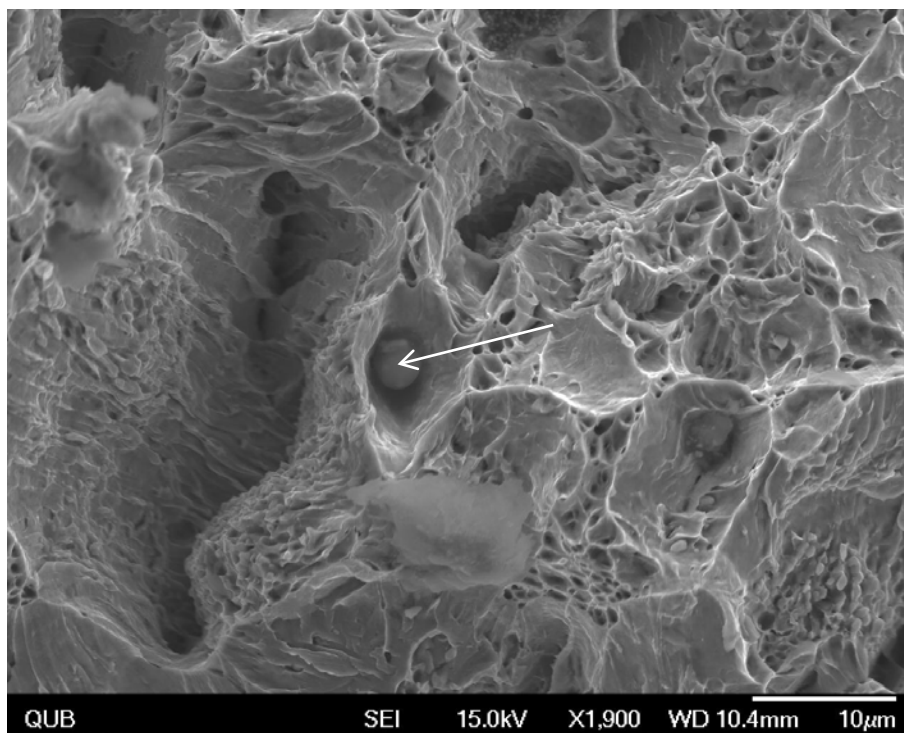


Fig. 5. Fracture surface of Fe-8.46%Mn-0.24%Nb-0.038%C, 850°C 1.5 h, WQ + 600°C 4 h, WQ, and then impact tested at -120°C, showing mainly ductile failure with cone-shaped dimples. Subsize impact energy 25 J. The particle pointed to by an arrow is an NbC precipitate, identified by EDS.

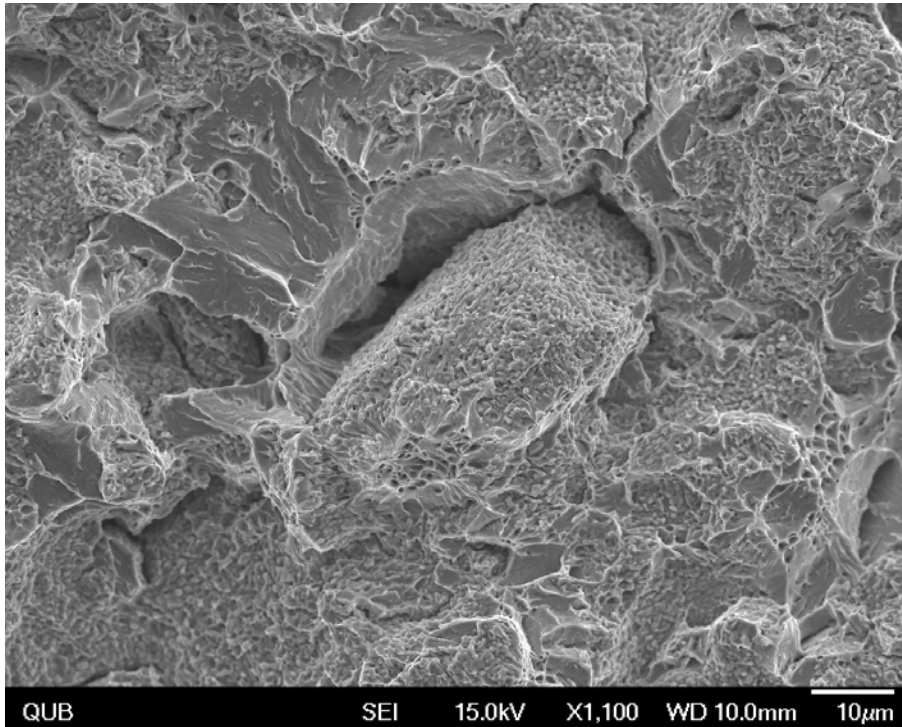


Fig. 6. Fracture surface of Fe-8.46%Mn-0.24%Nb-0.038%C, 850°C 1.5 h, AC + 600°C 4 h, AC, and then impact tested at -120°C, showing relatively brittle failure with a mostly honeycomb morphology. Subsize impact energy 15 J.

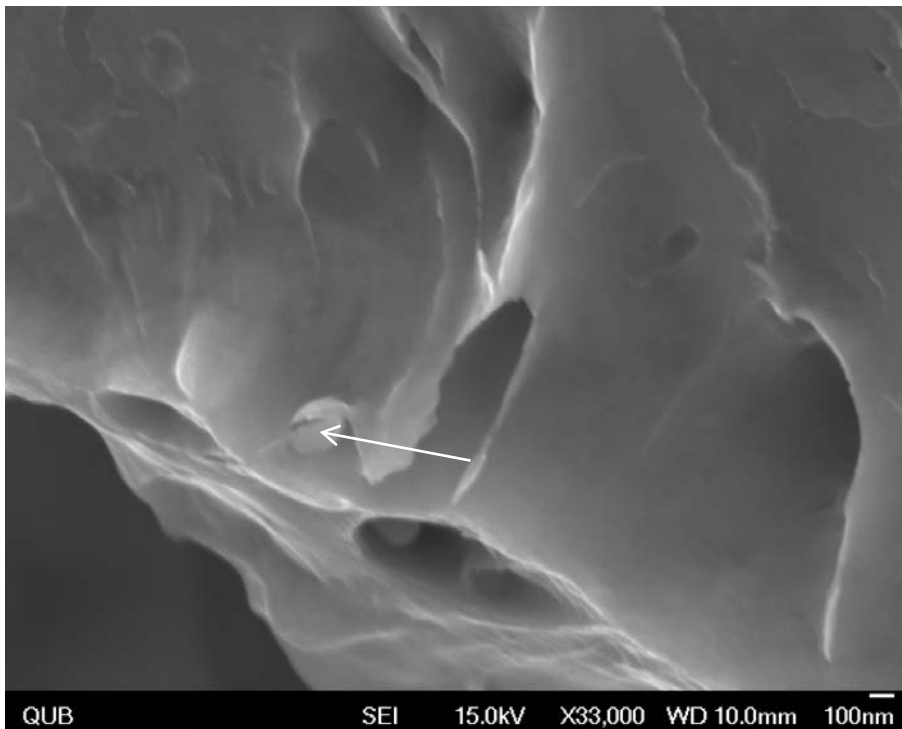


Fig. 7. Small crack propagating through NbC (identified by EDS).

CONCLUSIONS

The experimental results of the microstructure and hardness of the Fe-8.46%Mn-0.24%Nb-0.038%C manganese steel are summarised below.

- (1) The steel has a 100% bcc structure after 850°C 1.5 h, water quenching or air cooling. Austenite forms, in small amounts, after the 600°C reheating treatment. Deep quenching after these heat treatments does not change the phase composition.
- (2) Martensite interlocked microstructure consisting of fine martensite plates/needles with different spatial orientations was found in the steel.
- (3) NbC is formed in the steel, in high number densities.
- (4) The hardness of the steel after 850°C or 850°C followed by 600°C heat treatments is similar, at 265-280 HV. Deep quenching after these heat treatments does not change the hardness.
- (5) NbC plays a role in the impact fracture process, by acting as void nucleation sites, facilitating ductile fracture with dimples appearing on the fracture surface.

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